

# METHOD OF INCREASING THE HEAT INSULATING CAPACITY OF A MATERIAL

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## Abstract of WO 0208139 (A2)

A method of increasing the heat insulating capacity of a material such as a building component is achieved by including in the material a product which releases water at elevated temperatures. The product may be a water soluble crystallised compound in the form of a hydrate with two or more molecules of water, an inorganic hydrogel, or a combination of two or more hydrates or hydrogels, the hydrate or the hydrogel decomposing at a temperature in the range of from 40 DEG C to 500 DEG C inclusive to release water from the hydrate or the hydrogel to slow the progress of temperature elevation through the material.

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(54) Title: METHOD OF INCREASING THE HEAT INSULATING CAPACITY OF A MATERIAL

(57) Abstract: A method of increasing the heat insulating capacity of a material such as a building component is achieved by including in the material a product which releases water at elevated temperatures. The product may be a water soluble crystallised compound in the form of a hydrate with two or more molecules of water, an inorganic hydrogel, or a combination of two or more hydrates or hydrogels, the hydrate or the hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel to slow the progress of temperature elevation through the material.



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**METHOD OF INCREASING THE HEAT INSULATING CAPACITY**  
**OF A MATERIAL**

**BACKGROUND OF THE INVENTION**

This invention relates to a method of increasing the heat insulating capacity of a material, such as a finished product made from exfoliated vermiculite particles or expanded perlite particles. The finished product may be for example a panel or a board or the like.

For a building panel to be able to withstand the temperatures associated with a fully developed fire, i.e up to 1000°C, several attributes are required. These include dimensional stability, refractoriness, the absence of cracking and cohesive strength. However, above all, the thermal insulation properties of the building panel are important. By this is meant the resistance of the building panel to the transference of the heat of the fire through the body of the building panel, and the speed with which this occurs in fire.

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While various methods and means are known to increase the fire resistance of materials, there is always a need for new methods of this kind.

### **SUMMARY OF THE INVENTION**

According to a first aspect of the invention there is provided a method of increasing the heat insulating capacity of a material, including the step of including in the material a product which releases water at elevated temperatures selected from the group consisting of (i) a water soluble crystallised compound in the form of a hydrate with two or more molecules of water; (ii) an inorganic hydrogel; and (iii) a combination of two or more hydrates and/or hydrogels; the hydrate or the hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, with the proviso that the hydrate is not a hydrated hydraulic binder.

In one aspect of the invention, the product, i.e the hydrate or the hydrogel may be included in the material by impregnation as an aqueous solution, and subsequent drying.

For example, the method may include the steps of:

- (a) impregnating the material with an aqueous solution of:
  - (a1) a water soluble compound which when crystallised forms the hydrate; or
  - (a2) a compound which forms the hydrogel; or
  - (a3) a combination of (a1) and (a2); and
- (b) drying the impregnated material to induce crystallisation of the compound to form the hydrate or gelling of the compound to form the hydrogel, at a temperature below the decomposition temperature of the hydrate or the hydrogel.

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In step (a) the material is preferably provided in a substantially dry condition.

In step (b) the impregnated material is preferably dried to its equilibrium moisture content for the climate or environment in which it is to be used.

The material to be so impregnated and dried may be a formed product such as a panel, board, door or the like, or may be a starting material for such a formed product.

In a preferred method of this aspect of the invention, the material for use in step (a) is a cohesive product, the cohesive product being formed from a starting material by the steps of:

- (1) providing a feedstock selected from the group consisting of exfoliated vermiculite and expanded perlite or a mixture thereof, the feedstock optionally including an amount of expanded aluminium silicate, all in the form of substantially dry finely divided particles;
- (2) mixing the feedstock with
  - (2.1) a suitable amount of a thermosetting resin in finely divided dry powder form or in liquid form; and
  - (2.2) optionally a suitable amount of a hydraulic binder in finely divided dry powder form;to give the starting material; and
- (3) subjecting the starting material of step (2) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form the cohesive product.

In another preferred method of this aspect of the invention the material for use in step (a) is a rigid or a semi-rigid open cell thermoplastic or thermosetting foam element.

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In another aspect of the invention, the product, i.e the hydrate or the hydrogel may be included in the material by including in the material a precursor of the hydrate or the hydrogel, in dry form, and subsequently providing to the material water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.

For example, the method may include the steps of:

- (A) providing a starting material;
- (B) mixing the starting material with a suitable amount of the precursor;
- (C) forming the product of step (B) into a cohesive product; and
- (D) providing to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.

In a preferred method of this aspect of the invention, the starting material for use in step (A) is formed by the steps of:

- (1) providing a feedstock selected from the group consisting of exfoliated vermiculite and expanded perlite or a mixture thereof, the feedstock optionally including an expanded aluminium silicate, all in the form of substantially dry finely divided particles;
- (2) mixing the feedstock with:
  - (2.1) a suitable amount of a thermosetting resin in finely divided dry powder form or in liquid form; and
  - (2.2) optionally a suitable amount of a hydraulic binder in finely divided dry powder form;to give the starting material.

In this case steps (C) and (D) comprise:

- (C) subjecting the product of step (B) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form the cohesive product; and

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- (D) providing to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel, and for hydration of any hydraulic binder present to cause the hydraulic binder to set.

It is to be noted that the precursor must not decompose to give off gaseous products during step (C) which would result in failure of the product of step (B).

According to a second aspect of the invention there is provided a building component including a product which releases water at elevated temperatures selected from the group consisting of:

- (i) a water soluble crystallised compound in the form of a hydrate with two or more molecules of water;
- (ii) an inorganic hydrogel; and
- (iii) a combination of two or more hydrates and/or hydrogels;

the hydrate or the hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, with the proviso that the hydrate is not a hydrated hydraulic binder.

#### **DESCRIPTION OF EMBODIMENTS**

The crux of the invention is a method of increasing the heat insulating capacity of a material by including in the material a product which is either a water soluble crystallised compound in the form of a hydrate with two or more molecules of water, or an inorganic hydrogel, or a combination of two or more hydrates and/or hydrogels, the hydrate or hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, with the proviso that the hydrate is not a hydrated hydraulic binder.

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When a material treated by the method of the invention is subjected to a fire, as the material heats up above 40°C, depending upon the nature of the hydrate or the hydrogel in the material, water is released at a temperature or temperatures up to 500°C, either by being endothermically split from the hydrate or evaporated from the hydrogel, thereby removing the latent heat of vaporisation from the material and cooling it, until all the water is removed. This has the effect of slowing the progress of temperature elevation through the material.

In one aspect of the invention, the hydrate or the hydrogel may be included in the material by impregnation as an aqueous solution, and subsequent drying.

For example, the impregnation and drying may be carried out by:

- (a) impregnating the material with an aqueous solution of:
  - (a1) a water soluble compound which when crystallised forms the hydrate; or
  - (a2) a compound which forms the hydrogel; or
  - (a3) a combination of (a1) and (a2); and
- (b) drying the impregnated material to induce crystallisation of the compound to form the hydrate or gelling of the compound to form the hydrogel, at a temperature below the decomposition temperature of the hydrate or the hydrogel.

The material may be any material suitable for impregnation, in particular building elements such as panels, boards, doors and the like. Alternatively, the material may be a starting material for such a product. The preferred material is a cohesive product formed from exfoliated vermiculite or expanded perlite, a thermosetting resin, and optionally a hydraulic binder. Another preferred material is a rigid or a semi-rigid open cell thermoplastic or thermosetting foam element.



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Thus, in a preferred method of this aspect of the invention, there is provided a method of preparing a finished product from a cohesive product, the cohesive product being formed from a starting material including exfoliated vermiculite, expanded perlite, or a mixture thereof, a thermosetting resin and optionally a hydraulic binder.

Thus the first component used in forming the starting material is a feedstock comprising expanded perlite or exfoliated vermiculite or a mixture of the two, optionally also including an expanded aluminium silicate, in substantially dry finely divided particle form. In this regard, the particles preferably have a diameter of 0,5 mm or less. The preferred particle size ranges from 2 micron to 500 micron diameter inclusive.

An example of a suitable vermiculite is RSU submicron, with a particle size of less than 250 micron, by Phalaborwa Mining Company. Other examples are Zonolite No 4 and No 5 by W R Grace.

An example of a suitable perlite is Genulite M105 (having a bulk density of 105 g/l) or M75 (having a bulk density of 75 g/l) by Chemserve Perlite.

An example of a suitable expanded aluminium silicate is one with a bulk density in the range of from 150 to 220 g/l and a pH of from 6 to 7,5, such as ASP 170 by Engelhard.

The second component used in forming the starting material is a thermosetting resin in finely divided dry powder form or in liquid form.

By "finely divided" there is meant that the thermosetting resin must have a particle size of 98% passing a 200 mesh screen or finer (i.e a 74 micron screen or finer.)

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The thermosetting resin is preferably a novolac phenol formaldehyde resin with a suitable catalyst in finely divided dry powder form. Other thermosetting resins which may be used include isocyanate thermosetting resins, also known as polyurethane resins, together with their precursors such as MDIs, unsaturated polyester resins, phenol formaldehyde resole resins, epoxy resins, methacrylic acid ester resins, acrylic resins, urea formaldehyde resins, melamine formaldehyde resins and similar resins.

The thermosetting resin is preferably used in an amount of from 2% to 20% inclusive of the thermosetting resin by mass of the total mass of the feedstock, the thermosetting resin and the hydraulic binder, i.e total mass of the cohesive product before hydration.

The third optional component for use in forming the starting material is a hydraulic binder in finely divided dry powder form. A hydraulic binder is a substance which hydrates and sets in combination with water.

By "finely divided" there is meant that the hydraulic binder must have a particle size of 98% passing a 200 mesh screen or finer (i.e a 74 micron screen or finer). For example, Portland cement typically has a Blaine of 475, i.e 475m<sup>2</sup> per kg.

The hydraulic binder is preferably chosen from the group comprising Portland cement, high alumina cement, gypsum cement, calcium sulphate hemihydrate either in the alpha or beta form, magnesium oxychloride, magnesium oxysulphate, a calcium sulphotoaluminate cement, and pozzolans such ground granulated blast furnace slag, or a combination of any two or more thereof.

The hydraulic binder is preferably used in an amount of from 5% to 40% inclusive of the hydraulic binder, by mass of the total mass of the feedstock,

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the thermosetting resin and the hydraulic binder, i.e total mass of the cohesive product before hydration.

It is to be noted that certain hydraulic binders in themselves hydrate when exposed to water. In the method of the present invention, the hydraulic binder is used as a binder and in addition to the hydrate and not as a substitute therefor.

Other optional components may be included in the starting material, such as an amount of reinforcing fibres.

In the third step of the method of the invention, the starting material is subjected to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product.

For example, the starting material may be subjected to a temperature of from 125°C to 255°C inclusive and a pressure of from 10 to 70 kg/cm<sup>2</sup> inclusive.

In the next step (step (a)) of the method of the invention, the cohesive product is impregnated with an aqueous solution of a water soluble compound which compound either when crystallised forms a hydrate with two or more molecules of water, or forms a hydrogel, the hydrate or hydrogel decomposing at a decomposition temperature of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, the water being provided in an amount sufficient for the hydration of any hydraulic binder present to cause the hydraulic binder to set.

In the next step (step (b)) of the method of the invention, the product of the previous step is dried to induce crystallisation of the compound to form the hydrate or gelling of the compound to form the hydrogel, at a temperature below the decomposition temperature of the hydrate or the hydrogel.

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Suitable water soluble compounds which form hydrates with two or more molecules of water include the following:

Metal sulphate hydrates such as for example aluminium sulphate hydrate ( $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ); cadmium sulphate hydrate ( $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$  or  $\text{CdSO}_4 \cdot 4\text{H}_2\text{O}$ ); chromic sulphate hydrate ( $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$  or  $\text{Cr}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ) which loses  $12\text{H}_2\text{O}$  at  $100^\circ\text{C}$ ; sodium sulphate decahydrate, also known as Glauber's salt ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ); magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) which is a crystalline heptahydrate also known as Epsom salts, a solution of which has a pH of 6,8 and which loses  $6\text{H}_2\text{O}$  at  $150^\circ\text{C}$  and  $7\text{H}_2\text{O}$  at  $200^\circ\text{C}$ ; and zinc sulphate hydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) which loses  $5\text{H}_2\text{O}$  at  $70^\circ\text{C}$  eventually forming the anhydride at  $280^\circ\text{C}$ .

Disodium tetraborate decahydrate ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ), also known as borax, which is soluble in water and which loses  $8\text{H}_2\text{O}$  at  $75^\circ\text{C}$  and a further  $10\text{H}_2\text{O}$  at  $320^\circ\text{C}$ . This hydrate has a solids % of about 53% and a crystalline water % of about 47%.

A double salt of the formula



wherein M stands for a metal cation (including ammonium), and  $\text{M}^+$  and  $\text{M}^{3+}$  are different. The metal cation  $\text{M}^{3+}$  is preferably selected from the group consisting of aluminium, iron, chromium, manganese and cobalt, and the metal cation  $\text{M}^+$  is preferably selected from the group consisting of potassium, sodium, ammonium and cerium.

Examples of suitable double salts include:

aluminium potassium sulphate also known as potassium alum ( $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ ) which loses  $18\text{H}_2\text{O}$  at  $64,5^\circ\text{C}$ ; aluminium sodium sulphate also known as soda alum ( $\text{AlNa}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3 \cdot \text{Na}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ );

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chromium potassium sulphate also known as chrome alum ( $\text{CrK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ); aluminium ammonium sulphate also known as ammonium alum ( $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ ) which loses  $20\text{H}_2\text{O}$  at  $120^\circ\text{C}$ ;

chromium ammonium sulphate also known as chrome ammonium alum ( $\text{CrNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ); and

ferric potassium sulphate also known as iron alum ( $\text{FeK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ );

Sodium carbonate decahydrate also known as sal soda ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ );

Sodium metasilicate, which has the generic formula  $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ , and which loses  $6\text{H}_2\text{O}$  at  $100^\circ\text{C}$ . An example is Silchem 3379 by Silicate and Chemical Industries of South Africa, which as a typical solution has an SG of 1,395, a composition of 29,07%  $\text{SiO}_2$  and 8,81%  $\text{Na}_2\text{O}$ , a solid percentage of 37,88 in water, and a weight ratio of  $\text{SiO}_2$  to  $\text{Na}_2\text{O}$  of 3,3:1. Sodium metasilicate also has the advantage of being refractory and thereby contributing to the cohesive strength of the finished product.

Potassium silicate. This compound has a typical SG of 1,33, a composition of 23,86%  $\text{SiO}_2$  and 11,11%  $\text{K}_2\text{O}$ , a solid percentage of 35 in water, and a weight ratio of  $\text{SiO}_2$  to  $\text{K}_2\text{O}$  of 2.1:1 to 2.5:1. Potassium silicate has the advantage of being highly refractory and imposes an acid resistance to the final product.

Ferroso-ferric chloride ( $\text{FeCl}_2 \cdot 2\text{FeCl}_3 \cdot 18\text{H}_2\text{O}$ );

Dibasic sodium phosphate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ) which loses  $12\text{H}_2\text{O}$  at  $180^\circ\text{C}$ ;

Potassium pyrophosphate ( $\text{K}_4\text{P}_2\text{O}_7 \cdot 3\text{H}_2\text{O}$ ) which loses  $2\text{H}_2\text{O}$  at  $180^\circ\text{C}$  and  $3\text{H}_2\text{O}$  at  $300^\circ$ .

Magnesium orthophosphate ( $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$ ) which loses  $18\text{H}_2\text{O}$  at  $100^\circ\text{C}$ .

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Alumina trihydrate ( $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ).

The impregnating composition may include two or more suitable hydrates, for example, to give water release over a range of temperatures. For example, one may use a combination of potassium silicate both for its refractory properties and for its release of water, in combination with a compatible alum salt.

Another example of a suitable combination is magnesium sulphate and sodium borate which release water over a temperature range of from 75°C up to 330°C.

An example of a suitable hydrogel is a silica gel formed from a desiccated sodium silicate, the silica gel being hygroscopic and rapidly forming a saturated, stable, inorganic hydrogel on contact with water.

The water soluble compound or the compound which forms the hydrogel or the combination thereof is preferably dissolved in water in an amount of from 2% to 50% inclusive on a mass basis, depending upon the solubility of the compound in water.

The aqueous solution of the water soluble compound or the compound which forms the hydrogel may be impregnated into the cohesive product in any suitable manner. For example, the cohesive product may be placed in a pressure cylinder and optionally subjected to a pressure of from 10 to 15 kPa for 15 minutes. Thereafter the cylinder is flooded with the impregnating solution, and a pressure of from 200 to 600 kPa is applied for 10 minutes until penetration is complete. The impregnated cohesive product may then be removed from the pressure cylinder.

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Thereafter, the impregnated cohesive product is dried in a suitable kiln. Drying is preferably humidity drying with air flows in the region of 5 m/s to 8 m/s at temperatures preferably not exceeding 70°C.

It is to be noted that the material which has been impregnated must be dried at a temperature below that at which the hydrate decomposes to release the water of hydration or crystallisation or at which the hydrogel decomposes to release water, or at which the hydrate or hydrogel itself starts to decompose.

In another preferred method of this aspect of the invention the material for use in step (a) may be a rigid or a semi-rigid open cell thermoplastic or thermosetting foam element.

The foam element may be made of any suitable thermoplastic or thermosetting foam, including polyurethane, phenolic foams, silicone, polyimide and polyvinyl chloride.

The preferred foam is a polyurethane foam.

Polyurethane foams are generally formed from an isocyanate such as for example methyl phenyl di-isocyanate (MDI) or toluene di-isocyanate (TDI)

The polyurethane foams can be formed by reacting the isocyanates with hydroxyl compounds containing two or more reactive sites.

As indicated above, the foam must be a rigid or semi-rigid foam and must be open celled.

The size of the open cells is determined during the manufacture of the foam and is desirably in the range of from 1mm to 10mm inclusive in diameter, more preferably from 1mm to 3mm inclusive in diameter.

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A typical example of the manufacture of a polyurethane foam is the reaction of an MDI with a polyol in the presence of some water. The reaction between these compounds releases carbon dioxide which blows to form the foam.

The foam element may be any suitable foam element such as for example a length of foam for a wall insulating panel, or a foam tile for roof insulation or any other element utilised for insulation.

A foam element is preferably impregnated with an aqueous solution of an alkali metal silicate or a compound which forms a silica gel. After drying, a coating of the alkali metal silicate or the silica gel is left on a part or all of the surfaces of the cells of the foam element.

The alkali metal silicate may be a sodium silicate having a formula varying from  $\text{Na}_2\text{O} \cdot 3.75\text{SiO}_2$  to  $2\text{Na}_2\text{O} \cdot \text{SiO}_2$  and with various proportions of water.

The term sodium silicate is also intended to include sodium metasilicate e.g. sodium metasilicate pentahydrate, sodium sesquisilicate and sodium orthosilicate.

An example of a preferred sodium silicate is Silchem 3379 by Silicate and Industries as described above. This silicate is characterised by its propensity to intumesce or froth when subjected to fire.

The alkali metal silicate may also be a potassium silicate in which the weight ratio of  $\text{SiO}_2$  to  $\text{K}_2\text{O}$  varies from 2.1:1 to 2.5:1.

An example of a suitable potassium silicate is Silchem K2166 by Silicate and Chemical Industries which has a percentage of  $\text{SiO}_2$  of 23.86%, a percentage of  $\text{K}_2\text{O}$  of 11.11%, a solids percentage of 34.99% and a weight ratio of  $\text{SiO}_2$  to  $\text{K}_2\text{O}$  of 2.15:1.



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Alternatively, there may be used a silica gel. The silica gel is generally formed in situ in the foam element by reaction between an alkali metal silicate as described above, and an acid or an acidic gas.

The acid used is preferably a weak acid such as boric acid ( $\text{H}_3\text{BO}_3$ ) which reacts with sodium silicate to form a silica gel.

As an alternative to the use of an acid, an acidic gas such as carbon dioxide may be used to react with the alkali metal silicate to form the silica gel.

The coating may also include a crystalline hydrate which decomposes at a temperature of from  $40^\circ\text{C}$  to  $500^\circ\text{C}$  inclusive to release water from the hydrate.

In a first alternative, the solution of the alkali metal silicate which is impregnated into the foam element may contain a water soluble crystalline hydrate dissolved therein. On drying of the impregnated foam element, the water is removed, leaving behind the crystalline hydrate. An example of a suitable water soluble crystalline hydrate is magnesium sulphate,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  as described above.

In a second alternative, an insoluble crystalline hydrate may be dispersed, as very fine particles, in the solution of the alkali metal silicate. In this case, it is obviously important that the particle size of the crystalline hydrate is small enough to fit inside the cells of the open cell foam element. An example of a suitable insoluble crystalline hydrate is aluminium trihydrate,  $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ .

The use of a crystalline hydrate increases the water holding capacity of the insulating product, which is advantageous in fire.

The method of impregnating the foam element will now be described.

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The first step of the method is to impregnate the foam element with an aqueous solution of the alkali metal silicate. This solution may optionally contain a water soluble crystalline hydrate dissolved therein or a water insoluble crystalline hydrate dispersed therein.

The solids percentage in the aqueous solution is determined by the viscosity required of the solution, which in turn is dependent on the size of the open cells of the foam element.

The impregnation may be achieved for example by cascading the aqueous solution onto the foam element and allowing it to run through the foam element, thereby lining part or all of the surfaces of the open cells of the foam element with an alkali metal silicate film, the thickness of which is dependent on the viscosity of the solution.

Alternatively the foam element may be subjected to a vacuum in a cylinder, whereafter the cylinder is flooded with the aqueous solution.

The second step of the method of the invention is to remove any excess of the aqueous solution from the foam element. This may be achieved for example by allowing the excess aqueous solution to drain from the foam element, optionally assisted by a vacuum.

When it is desired that the foam element be coated with a silica gel, then the third step of the method of the invention is to impregnate the foam element with an acid or an acidic gas which reacts with the alkali metal silicate to form a silica gel.

The impregnation of the foam element with the acid or the acidic gas may be carried out before or after the impregnation of the foam element with the alkali

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metal silicate. In a preferred method, the foam element is first impregnated with the acid or the acidic gas and then with the alkali metal silicate.

As indicated above, a suitable acid is a weak acid such as boric acid. The boric acid reacts with the alkali metal silicate to form a silica gel which coats the surfaces of the open cells of the foam element.

The fourth step of the method of the invention is to remove any excess of the acid from the foam element.

Alternatively the foam element may simply be impregnated with an alkali metal silicate solution (without the use of an acid or acidic gas).

The fifth step of the method of the invention is to dry the product of the fourth step to give the insulating product.

The drying is preferably carried out with the application of heat, preferably not exceeding an initial temperature of 80°C, and dropping to 40°C as the excess water is driven off.

The result is an insulating product where the surfaces of the open cells of the foam element are coated with a coating of the alkali metal silicate or the silica gel, both of which have a cooling effect in fire by the release of water. In addition, and in particular with the silica gel, the coating tends to intumesce or froth at elevated temperatures, adding to the insulation and preventing a flame path in fire.

For example, an open cell polyurethane foam treated with a sodium silicate solution containing a crystalline hydrate, and preferably acidified to form a silica gel using either a weak acid such as boric acid or using an acidic gas such as carbon dioxide, can resist a fully developed fire for 90 minutes at a

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thickness of 50 mm with the temperature of an unexposed surface remaining below 110°C. In addition, such an insulating product, when used as an acoustic ceiling tile, has a good noise reduction co-efficient and is inexpensive.

The foam element may have a thin outer skin or skins placed thereon, either before or after coating with the alkali metal silicate or the silica gel.

For example, a polyurethane foam may be foamed between two thin outer skins to give a foam element which is then treated as described above.

As an alternative, the foam element may be produced and then have adhesively applied to one or more surfaces thereof a thin outer skin.

The thin outer skins may be made of any suitable materials such as for example:

Sheet metal skins, e.g steel or aluminium having a thickness of from 0.2 mm to 2 mm inclusive, which may be flat or surface embossed;

Dry pressed mineral skins having a thickness of from 1.5 mm to 12 mm, preferably from 1.5 mm to 6 mm inclusive, and with a density exceeding 1100 kg/m<sup>3</sup>, produced for example from expanded perlite or exfoliated vermiculite bound with an inorganic binder;

Pure vermiculite sheets having a thickness of from 1.5 mm to 6 mm inclusive and a density in the range of from 1300 kg/m<sup>3</sup> to 1700 kg/m<sup>3</sup>, formed from exfoliated vermiculite bound with a suitable resin such as for example a polyurethane resin or a phenol formaldehyde novolac resin;

Paper laminates, the paper being laminated using a suitable thermosetting resin such as a phenol formaldehyde resole resin or a polyurethane resin; and

Glass fibre reinforced polyurethane, polyester or phenol formaldehyde resole resin sheets.

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In the second aspect of the invention, the product, i.e the hydrate or the hydrogel may be included in the material by including in the material a precursor of a hydrate or a hydrogel, in dry form, and subsequently providing to the material water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.

For example, the method may include the steps of:

- (A) providing a starting material;
- (B) mixing the starting material with a suitable amount of the precursor;
- (C) forming the product of step (B) into a cohesive product; and
- (D) providing to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.

In a preferred method of this aspect of the invention, the starting material comprises exfoliated vermiculite or expanded perlite, a thermosetting resin, and optionally a hydraulic binder.

Details of suitable components for the starting material, viz. the exfoliated vermiculite, expanded perlite, thermosetting resin, and hydraulic binder are set out above.

There is included in the starting material a suitable amount of a precursor which is either an anhydrous or partially hydrous precursor of a compound which, when contacted with water, forms a hydrate with two or more molecules of water, or which is a precursor of a compound which, when contacted with water, forms a hydrogel, the hydrate or the hydrogel decomposing at a decomposition temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, the precursor not decomposing to give off gaseous products during formation of the cohesive product which would result in failure of the cohesive product, with the proviso that the precursor is not a hydraulic binder.

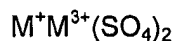
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Suitable precursor compounds for the hydrate include the following:

Metal sulphates such as for example aluminium sulphate ( $\text{Al}_2(\text{SO}_4)_3$ ) which hydrates to  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ; cadmium sulphate ( $\text{CdSO}_4$ ) which hydrates to  $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$  or  $\text{CdSO}_4 \cdot 4\text{H}_2\text{O}$ ; chromic sulphate ( $\text{Cr}_2(\text{SO}_4)_3$ ) which hydrates to  $\text{Cr}_2(\text{SO}_4)_3 \cdot 15\text{H}_2\text{O}$  or  $\text{Cr}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ; sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) which hydrates to sodium sulphate decahydrate, also known as Glauber's salt, i.e.  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ; magnesium sulphate ( $\text{MgSO}_4$ ) which hydrates to  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ; and zinc sulphate ( $\text{ZnSO}_4$ ) which hydrates to  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ .

Sodium borate, anhydrous ( $\text{Na}_2\text{B}_4\text{O}_7$ ) which hydrates to  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .

A double salt of the formula



wherein M stands for a metal cation (including ammonium) and  $\text{M}^+$  and  $\text{M}^{3+}$  are different. The metal cation  $\text{M}^{3+}$  is preferably selected from the group consisting of aluminium, iron, chromium, manganese and cobalt, and the metal cation  $\text{M}^+$  is preferably selected from the group consisting of potassium, sodium, ammonium and cerium. These double salts hydrate to give double salts of the formula

$\text{M}^+\text{M}^{3+}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ , examples being:

aluminium potassium sulphate also known as potassium alum ( $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ ) which loses  $18\text{H}_2\text{O}$  at  $64.5^\circ\text{C}$ ;

aluminium sodium sulphate also known as soda alum ( $\text{AlNa}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3 \cdot \text{Na}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ );

chromium potassium sulphate also known as chrome alum ( $\text{CrK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ );

aluminium ammonium sulphate also known as ammonium alum ( $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  or  $\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ ) which loses  $20\text{H}_2\text{O}$  at  $120^\circ\text{C}$ ;

chromium ammonium sulphate also known as chrome ammonium alum ( $\text{CrNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ ); and

ferric potassium sulphate also known as iron alum ( $\text{FeK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ );

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Sodium carbonate, anhydrous, i.e  $\text{Na}_2\text{CO}_3$ , or sodium carbonate monohydrate, i.e  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$  which hydrate to sodium carbonate decahydrate, also known as sal soda, i.e  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ;

Sodium metasilicate, anhydrous, i.e  $\text{Na}_2\text{SiO}_3$ , which hydrates to sodium metasilicate, i.e  $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$  which loses  $6\text{H}_2\text{O}$  at  $100^\circ\text{C}$ ;

Potassium silicate, anhydrous, which hydrates to potassium silicate.

Ferroso-ferric chloride, anhydrous, i.e  $\text{FeCl}_2 \cdot 2\text{FeCl}_3$ , which hydrates to  $\text{FeCl}_2 \cdot 2\text{FeCl}_3 \cdot 18\text{H}_2\text{O}$ ;

Dibasic sodium phosphate, anhydrous, i.e  $\text{Na}_2\text{HPO}_4$ , which hydrates to  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ;

Potassium pyrophosphate, anhydrous, ( $\text{K}_4\text{P}_2\text{O}_7$ ) which hydrates to potassium pyrophosphate  $\text{K}_4\text{P}_2\text{O}_7 \cdot 3\text{H}_2\text{O}$ ;

Magnesium orthophosphate, anhydrous ( $\text{Mg}_3(\text{PO}_4)_2$ ) which hydrates to magnesium orthophosphate  $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$ ;

reactive gamma alumina, i.e  $\text{Al}_2\text{O}_3$  which hydrates to  $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ .

A suitable precursor for a hydrogel is sodium silicate in dry powder form, e.g SP 33 by Silicate and Chemical Industries of South Africa, having a solids percentage of 84% and a weight ratio  $\text{SiO}_2$  to  $\text{Na}_2\text{O}$  of 3,3:1.

The anhydrous or partially hydrous precursor must not decompose to give off gaseous products during formation of the cohesive product. In other words, the precursor must not decompose nor give rise to any free water molecules which would cause an increase in pressure in the product being pressed, resulting in its destruction when the pressure is released.

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The anhydrous or partially hydrous precursor is preferably used in an amount of from 2% to 40% inclusive of the precursor by mass of the total mass of the feedstock, the thermosetting resin and the hydraulic binder, i.e total mass of the cohesive product before hydration.

In step (C) of the method, the starting material is subjected to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form a cohesive product. Suitable conditions are set out above.

In step (D) of the method, there is provided to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel, and for hydration of any hydraulic binder present to cause the hydraulic binder to set.

When reference is made to water, this is intended to include the use of liquid water as well as the use of water vapour or steam.

The water may be impregnated into the cohesive product by immersion of the cohesive product in water in a suitable chamber.

A preferred method of hydration is the use of low pressure steam or air saturated with water vapour, at temperatures in the range of from 40°C to 100°C inclusive, under pressure in a pressure cylinder, until the cohesive product has been subjected to sufficient water vapour to ensure the hydration of the precursor, and of any hydraulic binder present.

If necessary, any water in excess of that required in step (D) is removed, i.e the finished product is dried to the equilibrium moisture content.

Examples of the invention will now be given.



**Example 1**

A panel for the production of a fire rated door core was produced having a composition of 33% calcium sulphate hemi hydrate, 8% novolac phenol formaldehyde resin, and 59% expanded perlite of a particle size less than 0.5 mm diameter. The panel was pressed at a temperature of about 200°C to product a composite having a density of about 500 kg/m<sup>3</sup> at a thickness of 40 mm.

This dry composite was then impregnated with an aqueous solution containing 50% aluminium sulphate and 50% sodium sulphate, the sulphates being dissolved in the water in an amount of 15% by mass (combined sulphates).

The impregnated composite was then dried to a free water percentage of about 4%, i.e the water contained in the solution of the water soluble compound was substantially removed. The water of crystallisation or hydration formed when the aluminium sulphate and sodium sulphate crystallised was however retained within the impregnated composite.

The impregnated composite, when subjected to a fully developed fire with a furnace temperature exceeding 1000°C in a positive pressure furnace withstood the fire for 120 minutes, after which the side of the impregnated panel remote from the fire had a temperature not exceeding 110°C. Further, the integrity and degree of warpage in the composite panel remained substantially unchanged.

**Example 2**

A composition was formed as follows:

Exfoliated vermiculite particles	67%
Aluminium sulphate (Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> )	15%
Gypsum	10%
Resin – Novolac 602 by Schenectady Corporation	8%

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The composition was pressed to a density of about 450 kg/m<sup>3</sup> to give a cohesive product. The cohesive product was impregnated with water and then dried at 45°C at 15% relative humidity in a drier with an air speed of 6 m/s until the product reached a constant weight, to give the final product.

Tests were conducted on finished products made by the method of the invention compared to finished products not including a hydrate or a hydrogel. The products were as follows:

#### COMP 1 AND COMP 2

Comparative products comprising 28% gypsum, 8% resin and 64% vermiculite particles, were prepared by pressing at a defined temperature and pressure. The products were then impregnated with water only and dried.

#### VAS, PAS, PS, PA, VA, AND VS

Products of the invention comprising 28% gypsum, 8% resin and either 64% vermiculite particles (indicated by a V) or 64% perlite particles (indicated by a P) were prepared under the same conditions as the comparative products. The products were then impregnated with water containing 10% of aluminium sodium alum (AS) or sodium sulphate (S) or aluminium sulphate (A) and dried under the same conditions as the comparative products

The products were then analysed and the theoretical percentage gypsum, as if this was the only compound containing crystalline water present in the product, was calculated. The results are set out in Table 1.

**Table 1**

<b>Sample</b>	<b>% Free Water</b>	<b>% Mass Loss Due to Crystalline Water</b>	<b>Calculated % Gypsum</b>
COMP 1	5,25	-	22,5
COMP 2	8,7	-	22,0
VAS	2,0	7,0	34,0
PAS	1,4	6,8	33,0
PS	1,0	7,6	37,0
PA	0,9	7,4	36,0
VA	1,5	6,8	33,0
VS	2,4	5,7	28,0

It can be seen that in the products of the invention, the theoretical percentage of gypsum is significantly higher than for the comparative products, indicating an increase in the crystalline water, attributable to the presence of the hydrate compound.

In addition for the product VAS the crystalline water loss commenced at about 93°C whereas for the product COMP 1 the crystalline water loss only commenced at about 125°C.

### **Example 3**

A feedstock was prepared from:

Zonolite No 5 exfoliated vermiculite particles, by W R Grace	69%
Calcium sulphate $\beta$ -hemi-hydrate	30%
Novalac resin Code 602 by Schenectady	3%
12mm chopped strand fibre glass by Owens Corning	8%

The feedstock was pressed to a cohesive board having a density of about 500 kg/m<sup>3</sup> at 200°C.

After cooling, the cohesive board was post impregnated with a 12,5% solution by mass of magnesium sulphate in water.

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The resulting impregnated product was dried at 40°C by humidity drying to a free water percentage of 1,5% and a total percentage of water of crystallisation of 8,75%.

**Example 4**

A length of an open cell polyurethane foam with a cell size in the range of 1 mm to 3 mm inclusive was immersed in a 10% solution of boric acid in water. The excess solution was drained from the length of foam and the length of foam was partially dried.

Thereafter there was cascaded through the length of foam a 26% solids in water solution of sodium silicate (Code 3379 by Silchem). Excess of the solution was removed immediately. The length of foam was then dried at a temperature of about 60°C with an air speed of 6 m/s to form a rigid insulating product.

## CLAIMS

- 1 A method of increasing the heat insulating capacity of a material including the step of including in the material a product which releases water at elevated temperatures selected from the group consisting of:
  - (i) a water soluble crystallised compound in the form of a hydrate with two or more molecules of water;
  - (ii) an inorganic hydrogel; and
  - (iii) a combination of two or more hydrates and/or hydrogels;the hydrate or the hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, with the proviso that the hydrate is not a hydrated hydraulic binder.
- 2 A method according to claim 1 wherein the product is included in the material by impregnation as an aqueous solution and subsequent drying.
- 3 A method according to claim 2 including the steps of:
  - (a) impregnating the material with an aqueous solution of:
    - (a1) a water soluble compound which when crystallised forms the hydrate; or
    - (a2) a compound which forms the hydrogel; or
    - (a3) a combination of (a1) and (a2); and
  - (b) drying the impregnated material to induce crystallisation of the compound to form the hydrate or gelling of the compound to form the hydrogel, at a temperature below the decomposition temperature of the hydrate or the hydrogel.
- 4 A method according to claim 3 wherein the material for use in step (a) is a cohesive product, the cohesive product being formed from a starting material by the steps of:

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- (1) providing a feedstock selected from the group consisting of exfoliated vermiculite and expanded perlite or a mixture thereof, the feedstock optionally including an amount of expanded aluminium silicate, all in the form of substantially dry finely divided particles;
  - (2) mixing the feedstock with:
    - (2.1) a suitable amount of a thermosetting resin in finely divided dry powder form or in liquid form; and
    - (2.2) optionally a suitable amount of a hydraulic binder in finely divided dry powder form;to give the starting material; and
  - (3) subjecting the starting material of step (2) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form the cohesive product.
- 5 A method according to claim 2 wherein the material for use in step (a) is a rigid or a semi-rigid open cell thermoplastic or thermosetting foam element.
- 6 A method according to claim 1 wherein the product is included in the material by including in the material a precursor of the hydrate or the hydrogel, in dry form, and subsequently providing to the material water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.
- 7 A method according to claim 6 including the steps of:
  - (A) providing a starting material;
  - (B) mixing the starting material with a suitable amount of the precursor;
  - (C) forming the product of step (B) into a cohesive product; and
  - (D) providing to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel.

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- 8 A method according to claim 7 wherein the starting material for use in step (A) is formed by the steps of:
- (1) providing a feedstock selected from the group consisting of exfoliated vermiculite and expanded perlite or a mixture thereof, the feedstock optionally including an expanded aluminium silicate, all in the form of substantially dry finely divided particles;
  - (2) mixing the feedstock with:
    - (2.1) a suitable amount of a thermosetting resin in finely divided dry powder form or in liquid form; and
    - (2.2) optionally a suitable amount of a hydraulic binder in finely divided dry powder form;
- to give the starting material.
- 9 A method according to claim 8 wherein steps (C) and (D) comprise:
- (C) subjecting the product of step (B) to suitable conditions of temperature and pressure to cause the thermosetting resin to set to form the cohesive product; and
  - (D) providing to the cohesive product water in an amount sufficient for the hydration of the precursor to form the hydrate or the hydrogel, and for hydration of any hydraulic binder present to cause the hydraulic binder to set.
- 10 A method according to any one of claims 1 to 9 wherein the water soluble crystallised compound in the form of a hydrate with two or more molecules of water is selected from the group consisting of metal sulphate hydrates; disodium tetraborate decahydrate; a double salt of the formula  $M^+M^{3+}(SO_4)_2 \cdot 12H_2O$  wherein M stand for a metal cation and  $M^+$  and  $M^{3+}$  are different; sodium carbonate decahydrate; sodium metasilicate; potassium silicate; ferroso-ferric chloride; dibasic sodium phosphate; potassium pyrophosphate; magnesium orthophosphate; and alumina trihydrate.

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- 11 A method according to claim 10 wherein the metal sulphate hydrate is selected from the group consisting of aluminium sulphate hydrate, cadmium sulphate hydrate, chromic sulphate hydrate, sodium sulphate decahydrate, magnesium sulphate heptahydrate, and zinc sulphate hydrate.
- 12 A method according to claim 10 wherein the double salt is selected from the group consisting of aluminium potassium sulphate; aluminium sodium sulphate; chromium potassium sulphate; aluminium ammonium sulphate; chromium ammonium sulphate; and ferric potassium sulphate.
- 13 A method according to any one of claims 1 to 9 wherein the inorganic hydrogel is a silica gel formed from sodium silicate.
- 14 A method according to any one of claims 6 to 9 wherein the precursor of the hydrate is selected from the group consisting of a metal sulphate; sodium borate, anhydrous; a double salt of the formula  $M^+M^{3+}(SO_4)_2$  wherein M stands for a metal cation and  $M^+$  and  $M^{3+}$  are different; sodium carbonate, anhydrous; sodium metasilicate, anhydrous; potassium silicate, anhydrous; ferrous-ferric chloride anhydrous; dibasic sodium phosphate, anhydrous; potassium pyrophosphate, anhydrous; magnesium orthophosphate, anhydrous; and reactive gamma alumina.
- 15 A method according to claim 14 wherein the metal sulphate is selected from the group consisting of aluminium sulphate, cadmium sulphate, chromic sulphate, sodium sulphate, magnesium sulphate, and zinc sulphate
- 16 A method according to claim 14 wherein in the double salt of the formula  $M^+M^{3+}(SO_4)_2$ , the metal cation  $M^{3+}$  is selected from the group consisting of aluminium, iron, chromium, manganese and cobalt, and the metal



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cation  $M^+$  is selected from the group consisting of potassium, sodium, ammonium and cerium.

- 17 A method according to any one of claims 6 to 9 wherein the precursor of the hydrogel is sodium silicate in dry powder form.
- 18 A building component including a product which releases water at elevated temperatures selected from the group consisting of:
  - (i) a water soluble crystallised compound in the form of a hydrate with two or more molecules of water;
  - (ii) an inorganic hydrogel; and
  - (iii) a combination of two or more hydrates and/or hydrogels;the hydrate or the hydrogel decomposing at a temperature in the range of from 40°C to 500°C inclusive to release water from the hydrate or the hydrogel, with the proviso that the hydrate is not a hydrated hydraulic binder.
- 19 A building component according to claim 18 wherein the water soluble crystallised compound in the form of the hydrate with two or more molecules of water is selected from the group consisting of metal sulphate hydrates; disodium tetraborate decahydrate; a double salt of the formula  $M^+M^{3+}(SO_4)_2 \cdot 12H_2O$  wherein M stand for a metal cation and  $M^+$  and  $M^{3+}$  are different; sodium carbonate decahydrate; sodium metasilicate; potassium silicate; ferrous-ferric chloride; dibasic sodium phosphate; potassium pyrophosphate; alumina trihydrate and magnesium orthophosphate.
- 20 A building component according to claim 18 wherein the inorganic hydrogel is a silica gel formed from sodium silicate.